The Effect of Aggregate Size on the Interfacial Transition Zone in Concrete

by

Muhammad Muizzuddin Mohamad Izaha
4402

An Project Dissertation Submitted to
Universiti Teknologi PETRONAS
in partial fulfillment of the requirements for the
BACHELOR OF ENGINEERING (Hons)
(CIVIL ENGINEERING)

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CERTIFICATION OF APPROVAL

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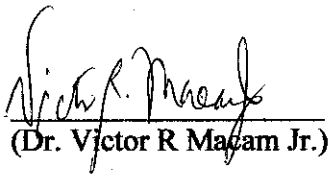

(Dr. Victor R Macam Jr.)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

December 2007

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD MUIZZUDDIN MOHAMAD IZAHA

ABSTRACT

This Final Year Project is about the effect of aggregate size on the interfacial transition zone in concrete. Concrete is a construction material that consists of cement, commonly Portland cement, aggregate (generally gravel and sand) and water. Concrete is strong in compression but it is extremely weak in tension. This project will study about the influence of interfacial transition zone towards concrete properties, the bond between aggregate and cement paste and the causes of weaknesses of concrete in tensile strength. Some experiment and test should be done in laboratory to analyze the performance of design mix concrete (high tensile strength concrete). Many researches and literature review also must be done to find the cause of weaknesses in tension and to recommend modification that can be made to improve tensile strength of concrete. The significance of this project is to obtain high performance concrete with exceptional high tensile strength and compressive strength for future construction and development. The result of this project also minimizes the cost for construction by reducing the need of steel reinforcement in structure.

ACKNOWLEDGEMENT

First and foremost, I would like to thank God for His guidance and help, for it is through His blessing that I am able to complete this Final Year Project. Thank you for guiding me towards completing this project.

Next I would like to express my heartfelt gratitude to my supervisor, Dr. Victor Rivera Macam Jr. He has done a tremendous job in monitoring and guiding me throughout the project. Thank you for giving me a chance to involve myself in the final year project under his supervision.

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CHAPTER 1

INTRODUCTION

1.1. Background of study

Concrete is the most widely used construction material in the world. Concrete is composed of cement, crushed rock or gravel, sand and water, often with chemical admixtures and other materials. These desired properties of concrete shall be met to make concrete useful:

- The concrete mix is workable (it can be placed and consolidated properly)
- Desired qualities of the hardened concrete are met (such as strength, permeability and resistance to thawing)
- Economical (this depends on water to cement ratio)

Concrete is preferred to be used worldwide because of its many advantages. Concrete is economical, durable and relatively low maintenance requirement and it is non-combustible material. It also has the ability to be cast into any desired shape. Concrete is a heterogeneous material consisting of three major components: bulk cement paste, aggregates, and interfacial transition zone (ITZ) (which is a thin shell with a thickness of 10 to 50 mm).

Concrete strength is directly related to the structure of hydrated cement (gel structure), water to cement ratio (w/c ratio) and the degree of compaction. A low w/c ratio and fully compacted concrete that means low porosity resulting a quality hardened concrete with good strength in both compression and tension. However, concrete has limitations. It is well known that concrete is very strong in compression, but it is extremely weak in tension. Normally, tensile strength of concrete is only around 10-15% of its compressive strength.

Tensile strength of concrete depends mostly on the shape and surface texture of aggregate, the bond between aggregate and cement paste, and the strength of interfacial transition zone (ITZ). The ITZ is considered to be weaker than either cement paste or aggregate; its microstructure is distinctly different from that of bulk cement paste in that its highly porous owing to higher water/cement ratio caused by the wall effect of the aggregate particles. Hence, ITZ has a profound influence on the mechanical behavior concrete.

1.2. Problem statement

Concrete is the most important material for construction. Concrete is very strong in compression, however, it is useless or extremely weak in tension. Concrete has relatively low tensile strength compared to the other building material. It has low ductility, low strength-to-weight ratio and cracking problems.

1.3. Significant of the project

By doing this project, the problem and the causes of concrete weaknesses in tension can be identify. A proper way to build high performance concrete that has high tensile strength can be done.

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1.4 Objective and Scope of Study

The main objective of this project is to study the effect of aggregate size towards ITZ, influence of interfacial transition zone towards concrete strength and its properties. The bond between aggregate and cement paste also being studied. Microstructures view of concrete and interfacial transition zone are obtained using scanning electron microscope (SEM).

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 What is Concrete

In its simplest form, concrete is a mixture of paste and aggregates. The paste, composed of portland cement and water, coats the surface of the fine and coarse aggregates. Through a chemical reaction called hydration [A.M.Neville [1]], the paste hardens and gains strength to form the rock-like mass known as concrete. Within this process lies the key to a remarkable trait of concrete: it's plastic and malleable when newly mixed, strong and durable when hardened. These qualities explain why one material, concrete, can build skyscrapers, bridges, sidewalks and superhighways, houses and dams.

The key to achieving a strong, durable concrete rests in the careful proportioning and mixing of the ingredients. A concrete mixture that does not have enough paste to fill all the voids between the aggregates will be difficult to place and will produce rough, honeycombed surfaces and porous concrete. A mixture with an excess of cement paste will be easy to place and will produce a smooth surface; however, the resulting concrete is likely to shrink more and be uneconomical. A properly designed concrete mixture will possess the desired workability for the fresh concrete and the required durability and strength for the hardened concrete. Typically, a mix is about 10 to 15 percent cement, 60 to 75 percent aggregate and 15 to 20 percent water. Entrained air in many concrete mixes may also take up another 5 to 8 percent

The main compound of Portland cement are tricalcium silicate (C3S), dicalcium silicate(C2S), tricalcium aluminate (C3A) and tetracalcium aluminoferrite. The composition of Portland cement is as shown in table 1. Particle size distribution of cement particles varied depends on the method of grinding. Most particle size of cement grains range between 3-30 μm.

Table 2.1: Cement Composition

Chemical Composition	% by mass
Loss On Ignition (L.O.I.)	1.29
Sulfur Trioxide (SO_3)	2.79
Silica Dioxide (SiO_2)	20.86
Ferric Oxide (Fe_2O_3)	3.47
Magnesium Oxide (MgO)	1.21
Aluminum Oxide (Al_2O_3)	4.60
Equivalent Alkalies (as Na_2O)	0.46
Calcium Oxide (CaO)	64.34
Free Lime	-
Insoluble Residue (I.R.)	0.1
Tricalcium Silicate (C_3S)	59.57
Tricalcium Aluminate (C_3A)	6.31

Portland cement's chemistry comes to life in the presence of water. Cement and water form a paste that coats each particle of stone and sand. The reactions by virtue of which Portland cement becomes a bonding agent take place in a water-cement paste. In the presence of water, the silicates and aluminates form products of hydration which in time produce a firm and hard mass – the hydrated cement paste [A.M.Neville [1]]. There are two

ways in which compound of the type present in cement can react with water that are through the true reaction of hydration and hydrolysis. The strength of concrete depends upon the hydration reaction. Hydration starts at the surface of the cement particles, thus it is the total surface area of cement that represents the material available for hydration. The hydration process is driven by three elements:

1. temperature
2. water
3. the availability of unhydrated cement

It has long been established that the water content of the concrete at the time of hardening plays a large role in determining the ultimate strength and durability of the concrete. It has also been reported that as the water content is increased the ultimate strength will decrease. This is correct because as the water content is increased, the particles of unhydrated cement are pushed further apart. This reduces the ability of the cement to bond to itself and to the aggregate. After concrete has begun its hardening process, the relative distances between the hydrated and unhydrated cement particles are by-and-large fixed. The introduction of water into the system at this point is not likely to cause a change in distances between the hydrated and unhydrated cement particles. The character of the concrete is determined by quality of the paste. The strength of concrete depends upon the hydration reaction.

H. Le Chatelier in 1882 states that the products of hydration of cement have a lower solubility than the original compounds, so that the hydrates precipitate from a supersaturated solution. The precipitate is in the form of interlaced elongated crystals with high cohesive and adhesive properties. Portland cement, when mixed with a large quantity of water, cement produces a solution supersaturated with $Ca(OH)_2$ and containing concentrations of calcium silicate hydrate in metastable condition R.H.Bogue [.2]. The calcium silicate hydrates are in the form of small (nanometers) interlocking crystals (gel) Bernal [3] which because of their size, could equally described as gel. When cement is mixed with a small quantity of water, the degree of crystallization is probably even poorer, the crystal being ill-formed.

The actual source of strength of gel arises from two kind of cohesive bonds T.C.Powers [4]:

- Physical attraction between solid surfaces, separated only by small(less than 3nm) gel pores. This is also refer as Van der Waals' force.
- Chemical bonds. The cement gel is of the limited swelling type. The gel particles are cross and linked by chemical forces. These forces are much stronger than Van der Waals' but only cover a small fraction of boundary of the gel particles.

The water-cement ratio is the weight of the mixing water divided by the weight of the cement. The strength of the cement paste depends on the ratio of water to cement. High-quality concrete is produced by lowering the water-cement ratio as much as possible without sacrificing the workability of fresh concrete. Generally, using less water produces a higher quality concrete provided the concrete is properly placed, consolidated, and cured. Water plays critical role (amount of water used). Water is added to give concrete sufficient workability. The water that was not consumed in hydration reaction will remain in the microstructure pore space. These pores make the concrete weaker due to lack of strength-forming calcium silicate hydrate bonds.

During the first week to 10 days of curing it is important that the concrete not be permitted to freeze or dry out because either of these, occurrences would be very detrimental to the strength development of the concrete. Theoretically, if kept in a moist environment, concrete will gain strength forever, however, in practical terms, about 90% of its strength is gained in the first 28 days. Typical strength-gain curve is shown in Figure 2.1. The industry has adopted the 28-day strength as a reference point, and specifications often refer to compression tests of cylinders of concrete which are crushed 28 days after they are made.

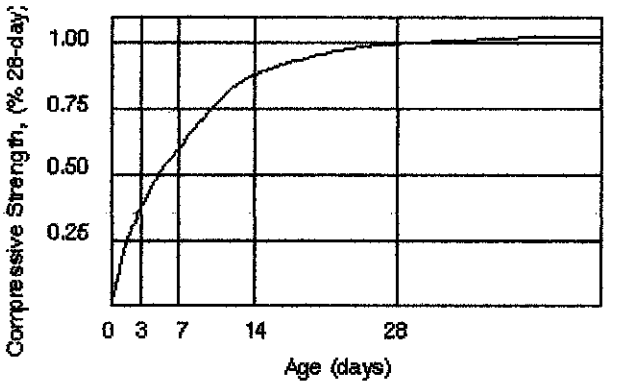


Figure 2.1: Typical strength-gain curve

Concrete has almost no tensile strength (usually measured to be about 10 to 15% of its compressive strength), and for this reason it is almost never used without some form of reinforcing. Its compressive strength depends upon many factors, including the quality and proportions of the ingredients and the curing environment. The single most important indicator of strength is the ratio of the water used compared to the amount of cement. Basically, the lower this ratio is, the higher the final concrete strength will be. (This concept was developed by Duff Abrams of The Portland Cement Association in the early 1920s and is in worldwide use today.) A minimum w/c ratio (water-to-cement ratio) of about 0.3 by weight is necessary to ensure that the water comes into contact with all cement particles (thus assuring complete hydration). In practical terms, typical values are in the 0.4 to 0.6 range in order to achieve a workable consistency so that fresh concrete can be placed in the forms and around closely spaced reinforcing bars.

Typical stress-strain curves A.M.Neville [1] for various concrete strengths are shown in Figure 2.2. Figure 2.2 indicates that the maximum strain that concrete can sustain before it crushes varies inversely with strength, a value of 0.003 is usually taken (as a simplifying measure) for use in the development of design equations.

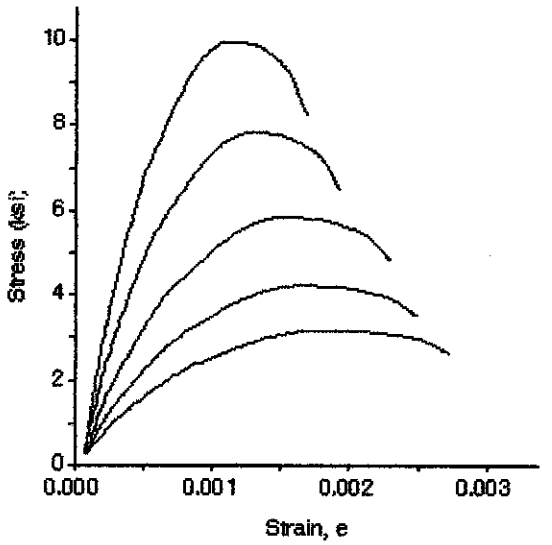


Figure 2.2: Stress versus Strain curves.

Because concrete has no linear portion to its stress-strain curve, it is difficult to measure a proper modulus of elasticity value. As concrete cures it shrinks because the water not used for hydration gradually evaporates from the hardened mix. For large continuous elements such shrinkage can result in the development of excess tensile stress, particularly if high water content brings about a large shrinkage. Concrete, like all materials, also undergoes volume changes due to thermal effects, and in hot weather the heat from the exothermic hydration process adds to this problem. Since concrete is weak in tension, it will often develop cracks due to such shrinkage and temperature changes. For example, when a freshly placed concrete slab-on-grade expands due to temperature change, it develops internal compressive stresses as it overcomes the friction between it and the ground surface. Later when the concrete cools land shrinks as it hardens) and tries to contract, it is not strong enough in tension to resist the same frictional forces. For this reason contraction joints are often used to control the location of cracks that inevitably occur and so-called temperature and shrinkage reinforcement is placed in directions where reinforcing has not already been specified for other reasons. The purpose of this reinforcing is to accommodate the resulting tensile stresses and to minimize the width of cracks that do develop.

In addition to strains caused by shrinkage and thermal effects, concrete also deforms due to creep. Creep is Increasing deformation that takes place when a material sustains a high stress level over a long time period. Whenever constantly applied loads (such as dead loads) cause significant compressive stresses to occur, creep will result. In a beam, for example, the additional long term deflection due to creep can be as much as two times the initial elastic deflection The way to avoid this increased deformation is to keep the stresses due to sustained loads at a low level. This is usually done by adding compression steel.

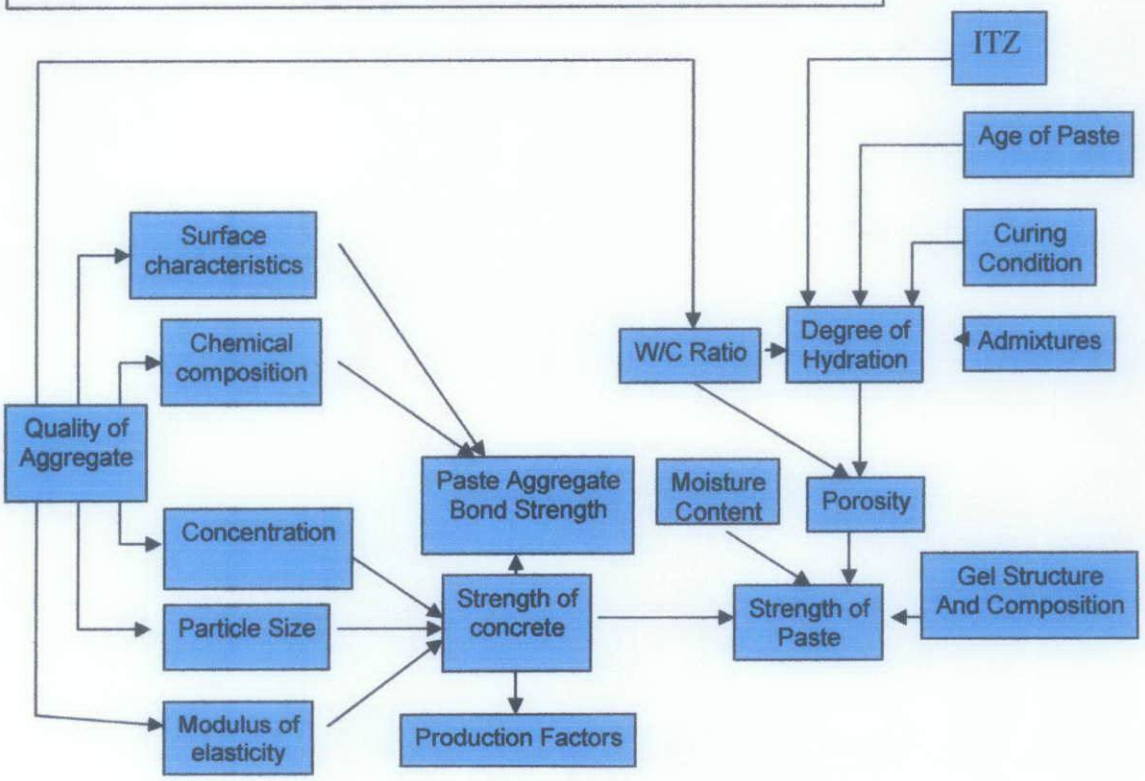
2.2 Factors affecting concrete strength

The strength of concrete at given age and cured in water at a prescribed temperature depends on two factors: the water–cement ratio and the degree of compaction. When concrete is fully compacted, its strength is taken to be inversely proportional to the water-cement ratio. Water-cement ratio determines the porosity of the hardened cement paste at any stage of hydration. Thus, the water-cement ratio and the degree of compaction both affect the volume of voids in concrete Gilkey [5]. Strictly speaking, strength of concrete is influenced by the volume of all voids in concrete; entrapped air, capillary pores, gel pores and entrained air, if present. Porosity in concrete can be defined as the total volume of the overall volume of pore larger than gel pores. Porosity without doubt is a primary factor influencing the strength of the cement paste. Smaller pore leads to a higher strength of the cement paste. The water not consumed in the hydration reaction will remain in the microstructure pore space. These pores make the concrete weaker due to the lack of strength-forming calcium silicate hydrate bonds. Some pores will remain no matter how well the concrete has been compacted.

The actual strength of hydrated cement paste or a similar brittle material such as stone is very much lower than the theoretical strength estimated on the basis of molecular cohesion, and calculated from the surface energy of a solid assumed to be perfectly homogenous and flawless. This discrepancy can be explained by the presence of flaws postulated by Griffith Griffith [7]. These flaws lead to high stress concentrations in the

material under load so that a very high stress is reached in very small volumes of the specimen with a consequent microscopic fracture, while the average nominal stress in the whole specimen is comparatively low. Hydrated cement paste is known to contain numerous discontinuities (pores, microcracks and voids) but the exact mechanism through which they affect the strength is not known. The relation between factors affecting concrete strength is shown in Figure 2.3.

Figure 2.3: Factors affecting concrete strength and relationship between them



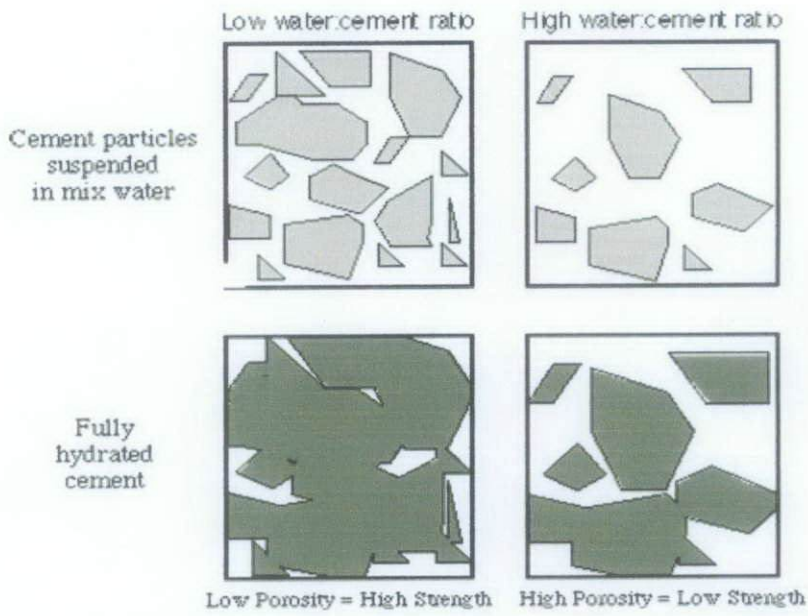


Figure 2.4: Schematic drawings to demonstrate the relationship between the water/cement ratio and porosity.

The empty space (porosity) is determined by the water to cement ratio. The relationship between the water to cement ratio and strength is shown in the graph that follows. Low water to cement ratio leads to high strength but low workability. High water to cement ratio leads to low strength, but good workability.

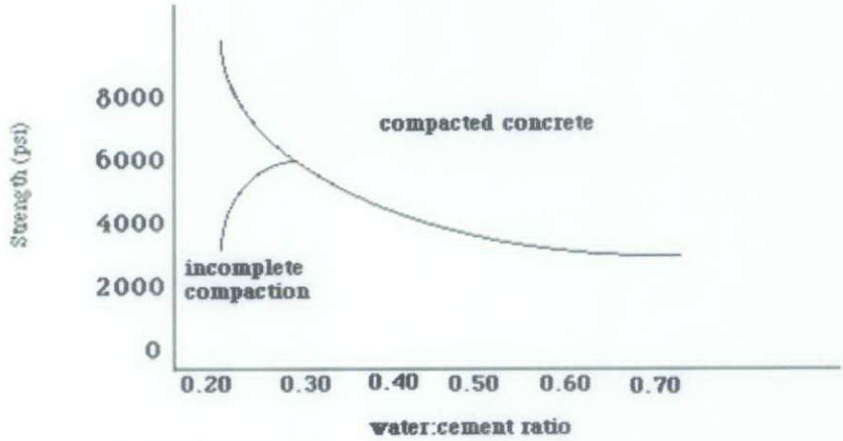


Figure 2.5: A plot of concrete strength as a function of the water to cement ratio.

The physical characteristics of aggregates are shape, texture, and size. These can indirectly affect strength because they affect the workability of the concrete. If the aggregate makes the concrete unworkable, the contractor is likely to add more water which will weaken the concrete by increasing the water to cement mass ratio.

Time is also an important factor in determining concrete strength. Concrete hardens as time passes. This is because the hydration reactions get slower and slower as the tricalcium silicate hydrate forms. It takes a great deal of time for all of the bonds to form which determine concrete's strength. It is common to use a 28-day test to determine the relative strength of concrete.

The tensile strength of concrete can be measured by radically different tests, namely flexure, direct tension, and splitting, and the resulting values of strength are not the same. The tensile strength of concrete is more sensitive to inadequate curing than the compressive strength possibly because the effect of non-uniform shrinkage of flexure test beams are very serious. Thus, air-cured concrete has a lower ration than concrete cured in water and tested wet.

There are several factors that affecting tensile strength of concrete Neville [1] :

- Shape and surface texture of aggregate
    - a rougher texture result in a larger adhesive force between the particles and the cement matrix
    - larger surface area of angular aggregate contribute to larger adhesive force
- Bond between aggregate and cement paste
    - Bond is due, in part, to the interlocking of the aggregate and the hydrated cement paste due to the roughness of the surface of the former.
    - Rougher surface (crushed particles) gives better bond due to mechanical interlocking

- Better bond also obtained with softer, porous and mineralogically heterogeneous particles
- Bond also affected by other physical and chemical properties of aggregate, related to its mineralogical and chemical composition, and to the electrostatic condition of the particle surface

- The Interfacial Transition Zone (ITZ)
  - The interface zone has higher porosity than the hydrated cement paste and the coarse aggregate. High porosity causes low strength of concrete
  - Wall effect causes cement particles unable to become closely packed against the relatively large particles of aggregate.
- The influence of aggregate on flexural strength also depends on the moisture condition of the concrete at the time of test.
- Impurities in mixing water and aggregate – More additive material causes decreasing in both tensile and compressive strength.

2.3 Interfacial Transition Zone (ITZ)

Properties of concrete should be analyzed by considering it as a three-phase composite: aggregate, ITZ, and cement paste. In fresh concrete, water:cement (w/c) ratio gradient develops around the aggregate particles during casting, resulting in a different microstructure of the surrounding hydrated cement paste. This zone around the aggregate is called the interracial transition zone (ITZ). According to numerous researchers, the thickness of ITZ is 40-50 μm. The higher w/c ratio implies a diffusion process during hydration, and this zone may be consequently described as a heterogeneous area with a porosity gradient and a complementary gradient of anhydrous and hydrated phases.

In freshly compacted concrete, water films form around the large aggregate particles. This would account for a higher w/c ratio, closer to the larger aggregate than away from it. As in the bulk paste, calcium, sulfate, hydroxyl and aluminate ions are produced by

the dissolution of calcium sulfate and calcium aluminate compounds, and are then combined to form ettringite and calcium hydroxide (CH) [J. C. Maso[8]]. Owing to the higher w/c ratio, these crystalline products in the vicinity of the coarse aggregate consist of relatively larger crystals. And, therefore, they form a more porous framework than in the bulk cement paste or mortar matrix. With the progress of hydration, poor crystalline C-S-H and a second generation of smaller crystals of ettringite and CH crystals start filling the empty space that exists between the framework created by the large ettringite and CH crystals. This helps improve the density and, hence, the strength of the transition zone.

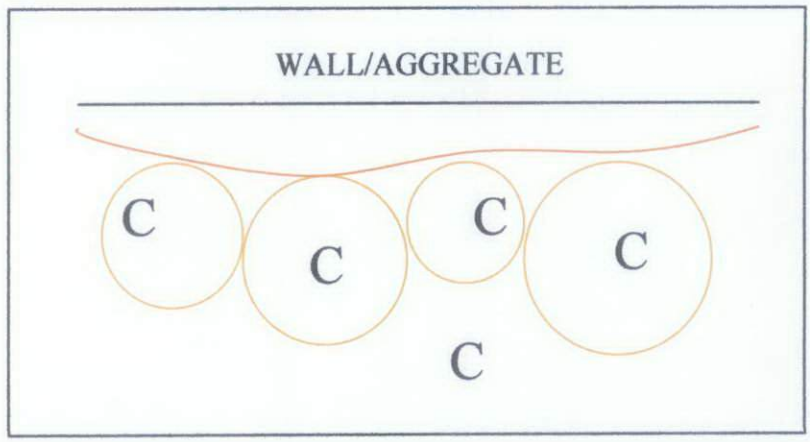


Figure 2.6: Wall effect phenomenon. C represents cement grains while red line represents region at the interface which appears to be devoid of particles.

During mixing or casting, dry cement particles are unable to become closely packed against relatively large particles of aggregate. Aggregate is effectively a wall against which the cement particles must pack themselves. Even though the particles are touching, the amount of empty spaces approaches 100% as the wall is approached. This phenomenon is known as wall effect (Figure 2.6). Wall effect phenomenon prevent effective filling of the space adjacent to the aggregate with cement grains having a size of 10μm or more. As a result, the space around the aggregates is less effectively filled by hydration products, and at the same time, there is greater tendency for CH and ettringite to develop. The so-called wall effect has been extensively presented to explain

the special features of the ITZ. During casting, the spatial arrangement of anhydrous grains becomes looser in the vicinity of aggregate particles. Consequently, in fresh concrete the porosity and the w/c ratio increase from the bulk to the surface of aggregate particles. A complementary explanation has been proposed: during the vibration of concrete and before setting, microbleeding leads to an accumulation of water under the aggregate particles. Due to the first effect, the fresh cement paste which surrounds the aggregate grains exhibits a gradient of water and a complementary gradient of anhydrous cement. Due to the second effect, certain heterogeneity of the gradient around the particles may be foreseen. Thus, wall effect happen and there is less cement present to hydrate and fill the original voids. In consequence, the interface zone has a much higher porosity than the hydrated cement paste and the coarse aggregate. High porosity causes weakness in concrete.

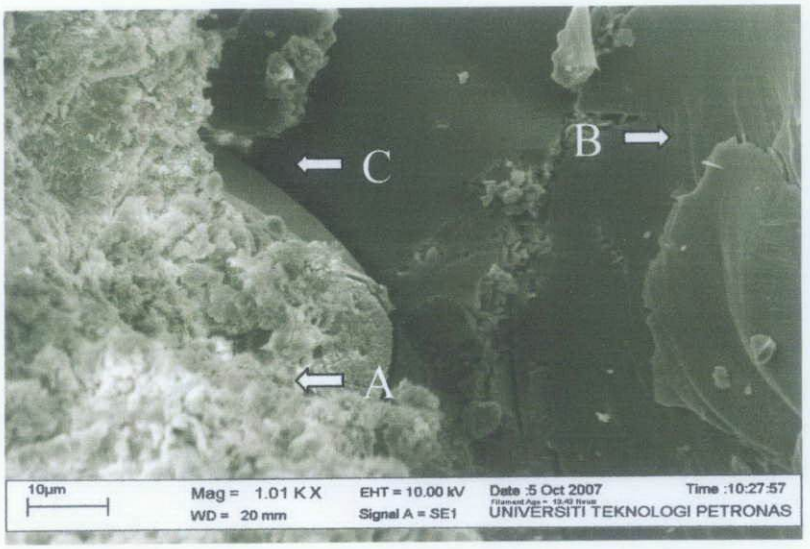


Figure 2.7: Interfacial microstructure around an aggregate in ordinary Portland cement concrete: (A) the interfacial transition zone; (B) aggregate ; (C) gap between aggregate and interface due to wall effect phenomenon

The microstructure of ITZ is as follows. The surface of the aggregate is covered with a layer of oriented crystalline $Ca(OH)_2$, about 0.5 µm thick, behind which there is a layer

of C-S-H of about the same thickness. This is referred to as duplex film. Further away from the aggregate, there is the main interface zone, some 50 μm thick, containing products of hydration of cement with larger crystals of $Ca(OH)_2$ but without any unhydrated cement [L.A.Larbi [7]].

The significance of the above distribution is twofold. First, the complete hydration of cement indicates that the w/c ratio at the interface is higher than elsewhere. Seconds, the presence of large crystals of $Ca(OH)_2$ indicates that the porosity at the interface is higher than elsewhere, this confirms the wall effect earlier.

The microstructure may be described in terms of the porous microstructure and the hydration progress. The two approaches are obviously linked. Hydration in the vicinity of aggregate grains differs from the reaction which take place in the bulk paste because the w/c ratio is locally higher and because the growth and the nature of hydrates may be influenced by the surface and the chemical nature of aggregates. So the excess of porosity is both the cause and the consequence of the existence of the ITZ.

The quantification of the porosity in the ITZ has been performed by means of two main techniques: image analysis of flat polished surfaces observed by SEM using back-scattered imaging, and mercury intrusion porosimetry (MIP). The first technique gives local data, whereas the second gives bulk information. Through SEM, porosity of ITZ can be observed. The observation is made on flat sections of mortars or concretes allow one to determine the porosity as a function of the distance to the aggregate surface. Because the pore size measured in the two-dimensional images does not have a stereological nature, it is not possible to obtain the three-dimensional pore-size distribution using this technique.

With young OPC paste the porosity increases near the surface of the aggregate particles and the thickness of this disturbed area is 15 μm, the order of magnitude of the cement grain diameter. The main variations in porosity occur in the first 15 to 20 μm. The variation of the porosity decreases with age due to filling by hydrates. Two conclusion

can be made about porosity in ITZ; (1) the porosity is higher in the ITZ than in the bulk (two to three times more); and (2) the pores are coarser in the ITZ. These features can be explained by a poor arrangement of cement grains in the vicinity of the aggregate particles.

The hydration of the cement and the resulting features in the ITZ may be described by considering diffusion of ions and spatial constraints. As large areas filled with water are available in the vicinity of the aggregate particles, the hydration happens mainly following the Le Chatelier mechanism. Then, after the dissolution of anhydrous compounds, the more mobile ions move under the influence of concentration gradients (i.e., from the bulk to the interface. In OPC, Na ÷ , K +, SO42-,Al(OH)4 -, and CA 2+ ions diffuse faster). The mobility of silicate ions is lower and they diffuse more slowly from the silicate grains to the aggregate surface. The evolution of the profiles between 28 days and 1 year is not similar for the two hydrates: the diffusion of calcium ions seems to be more influenced by the packing in the ITZ during hydration than the silicate ions do.

Another factors affecting ITZ is aggregate. Size of aggregate largely affects the formation of products at the surface. A large aggregate probably favors the formation of a continuous layer of water at the surface which would in turn favor the formation of CH. Whereas, in smaller size of aggregate, C-S-H would be most likely to precipitate on the aggregate surface. The microstructure in the ITZ also may be affected by a chemical reaction of the aggregate with the cement paste such as the reaction between calcerous aggregate with OPC paste. The hydrate which reacts is calcium aluminate hydrate, forming calcium carbo-aluminate (CsA.CaCOs.11 H20). This reaction is limited to the interfacial zone due to the low mobility of carbonate ions. The reaction leads to an improvement of the compactness of the paste.

2.4 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) as shown in Figure 2.8 permits the observation and characterization of heterogeneous organic and inorganic materials on a nanometer (nm) to micrometer (μm) scale. A major reason for the SEM's usefulness is the high resolution which can be obtained when bulk objects are examined; instrumental resolution on the order of 1-5 nm (10-50A) is now routinely quoted for commercial instruments. Another important feature of the SEM is the large depth of field, which is responsible, in part, for the three-dimensional appearance of the specimen image. The basic components of the SEM are the lens system, the electron gun, the electron collector, the visual and photorecording cathode ray tubes (CRTs), and the associated electronics Joseph Goldstein[9].

SEM has been used in two different modes of imaging: *secondary electron imaging* (SE) to study fracture surfaces where topographical features produce the contrast in the image and the large depth of focus is valuable, and *back-scattered electron imaging* (BSE) to study flat polished surfaces where differences in back-scattering coefficient, dominated by atomic number differences, produce the contrast. SE are loosely bound outer shell electrons from the specimen atoms which receive sufficient kinetic energy during inelastic scattering of the beam electrons to be ejected from the atom and set into motion. The SE thus created will propogate through the solid, and some will intersect the surface and escape. SE are defined purely on the basis of their kinetic energy. BSE are beam electrons whose trajectories have intercepted a surface, usually, but not necessarily, the entrance surface and which thus escape the specimen. Generally, these electrons have undergone numerous elastic scattering events to accumulate enough deviation from the incident beam path to return to the surface. BSE remove a significant amount of the total energy of the primary beam, which in the absence of the backscattering effect would contribute to the production of additional secondary radiation products such as the characteristics x-rays measured in quantitative x-ray microanalysis.

In the SEM, high energy electrons are focused into a fine beam, which is scanned across the surface of the specimen. Complex interactions of the beam electrons with the atoms of the specimen produce a wide variety of radiation products: backscattered electrons, secondary electrons, absorbed electrons, characteristic and continuum x-rays. A sample of this radiation is collected by a detector and the collected signal is amplified and displayed on a cathode ray tube (CRT) or television screen scanned in synchronism with the scan on the specimen. The resulted image is somewhat easy to interpret.

Figure 2.8: Scanning Electron Microscope

CHAPTER 3

METHODOLOGY AND PROJECT WORK

3.1 Literature Review and Information Gathering

Information gathering can be done via research through internet, article, journal and books from Information Resource Centre. Besides, the information also gathered by discussion and consulting lecturers for better understanding.

3.2 Familiarization of the Tools / Equipment

All tools / equipment are available in Civil Department concrete lab except for scanning electron microscope (SEM). With the cooperation and appointment with Mechanical Department technician, the SEM can be used. In the future, if any additional equipment needs to be used, a discussion with the supervisor will be conducted.

3.3 Experimental and Result Analysis

First, the aggregates need to be sieved into different size of aggregates that is 10, 14 and 20 mm. Sand that being used for mixing also being sieved and the particle distribution curve was plotted. This is to know whether the sand was well graded or not. Figure 11 below shows the aggregates that have been sieved according to the respective required size.

Figure 3.1: Aggregate that has been sieved and arranged according to the size.

To measure the tensile stress, split tensile test and flexural test were conducted. For split tensile test, three batch of mix were done. Figure 5a and 5b show the machine that being used for the test.

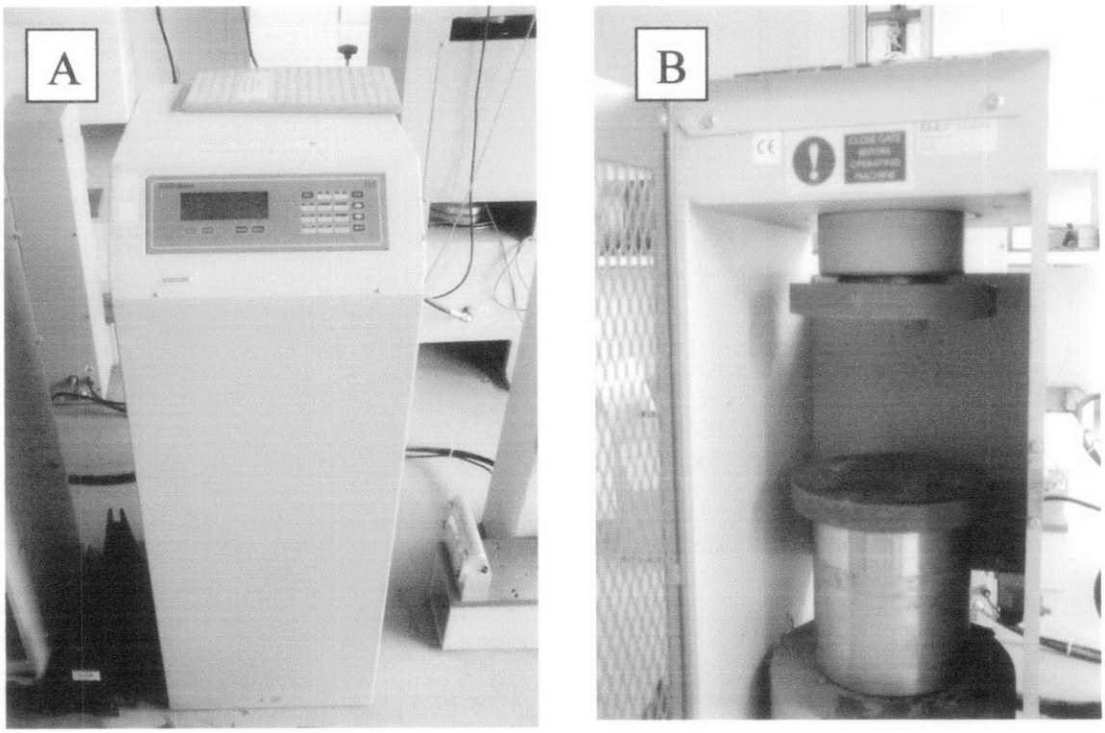


Figure 3.2: Equipment used for tensile strength test; (A) Flexural and Compression machine for split tensile test. (B) Flexural and Compression Machine for split tensile test.

9 cubes of 100 x 100 x 100mm and 3 prism of 100 x 100 x 500mm were used for each mix. W/C ratio used for every mix is 0.5. Each of the mix only uses one particular size of aggregate. The size of aggregate used was 10mm, 14mm and 20mm. The mix proportions for the concrete mix are listed in the table 1, 2 and 3. Tensile strength using split tensile test was measured by using the cube specimens at 3, 7 and 28 days of strength. For flexural test, the prism was tested at 28 days of strength.

Table 3.1: Concrete mix proportion for 10 mm aggregate

Material	Weight (kg)
Water	7.9
Cement	15.2
Fine Aggregate	24.51
Coarse Aggregate	33.84

Table 3.2: Concrete mix proportion for 14 mm aggregate

Material	Weight (kg)
Water	7.48
Cement	14.95
Fine Aggregate	21.55
Coarse Aggregate	33.7

Table 3.3: Concrete mix proportion for 20 mm aggregate

Material	Weight (kg)
Water	6.84
Cement	13.16
Fine Aggregate	21.3
Coarse Aggregate	41.35

3.4 Scanning Electron Microscope Application

For microstructure and interfacial transition zone studies, small samples need to be prepared. Cube needs to be break into smaller particles in range size of 5-25mm. Selected 3, 7 and 28 days are chosen for sample preparation for every mix. Samples need to be dry in order to remove free water in the specimen and avoid hydration. These samples were then examined using Scanning Electron Microscope (SEM) (See Figure 2.8).The development of microstructure and interfacial transition zone in concrete can be observed through SEM. For each sample, 3 SEM pictures were taken from different view of interfacial zone for comparison and better understanding.

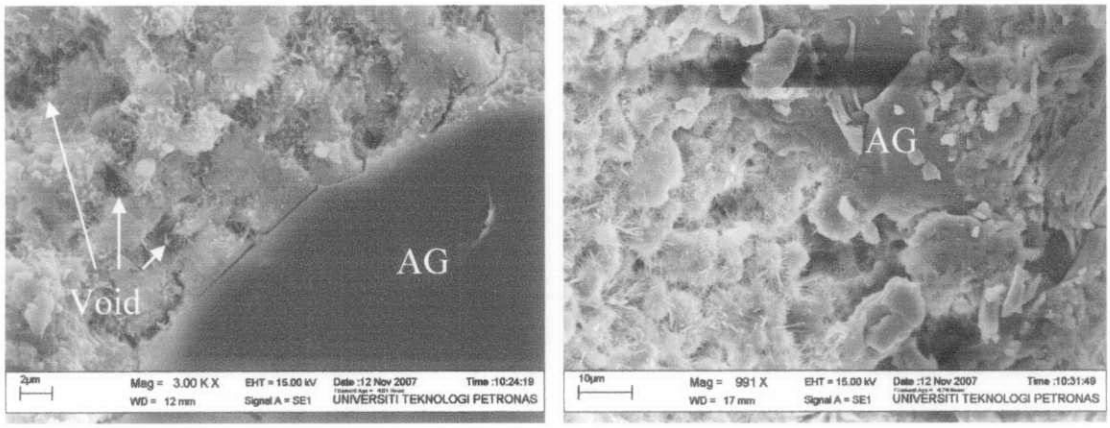
CHAPTER 4

RESULT AND DISCUSSION

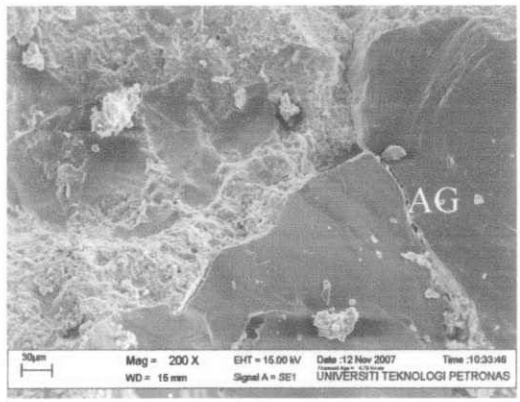
4.1 Effects of Ageing on the ITZ

Starting with freshly mixed concrete in the plastic state, the ITZ changes from predominantly a water-filled zone around an aggregate particle to a zone increasingly filled with solids and whose porosity reduces with time. Rates of deposition and growth of solids in this zone will differ from those in the bulk paste. In the region of high local w/c ratio around the aggregates, ions produced by dissolution of cement grains accumulate by migration to form a solution supersaturated in calcium and hydroxyl ions. Thus the first effect is that of a precipitate of CH forming as a coating on the aggregate surface [J. C. Maso]. The next phase occurs when the sulphate, aluminate and silicate ions have also passed into the solution phase, and then together with other calcium and hydroxyl ions, precipitate out as CH, ettringite and C-S-H. The CH crystals tend to be large due to large space available. It is important to notice that it is the presence of large quantities of CH that causes the weak of mechanical strength of the ITZ.

Figure 4.1 shows that as the time go on, initial interfacial zone that was full with voids and gaps partially filled with CH crystals or porous hydration becomes denser. The porosity also reduced after 28 days by observation of number of pores that can be observed. There is not so much different between 3 days and 7 days because the time gap is quite close. Both have many voids and large CH crystals together with ettringites and C-S-H products. At 28 days old, the densification of ITZ with time due to continuing deposition of hydration products clearly apparent. Thus, the microstructure view of ITZ of 14 mm mix clearly looks denser with lesser pore. The size of ITZ does no change with time, although the orientation index of CH in the zone does change markedly with time.



(a) (b)



(c)

Figure 4.1: ITZ for 14 mm mix (w/c = 0.5). The aggregate particle is marked by *AG*.
(a) 3 days old (b) 7 days old (c) 28 days old

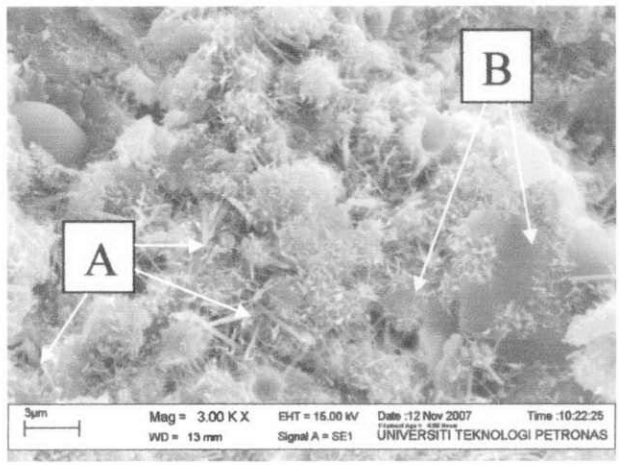


Figure 4.2: Microstuctural view of ITZ; (A) C-S-H products
(B) CH crystals

At age of 3 days and 7 days (Figure 4.1 (a) and (b)), a few C-S-H gels can be found, but the hydration products of C-S-H increase quickly with the increase in curing age. The hydration products of C-S-H appeared as needle-like shape (Figure 4.2), and the other hydration products such as ettringite and CH also exists. The needle-shaped C-S-H increased and gradually formed the gel, with the needles growing into pore space just like mesh. At age of 28 days (figure 4.1(c)), there were still much hydration products caused by the pozzolanic reaction. Although there still many pores can be found, some of the previous pores were now filled with hydration products, making the previous mesh structure turn into plan structure. Pores also found to be decreased, however, still more than in bulk paste.

Many authors have commented on chemical reactions occurring with time between the cement paste phase and aggregates. Many of these are claimed to be beneficial to the bond between paste and aggregate, but some authors point to chemical reaction actually weakening the bond strength. Excessive chemical dissolution of marble ($CaCO_3$) by the liquid phase of the adjoining cement paste may weaken the bond by precipitating large amounts of CH in the form of large crystals in the interface. As stated before, the presence of CH will reduce the strength of ITZ.

Bond strength is taken to refer to the cleavage or tensile-type adhesion of paste to an aggregate particle. Bond strength will depend on several factors; type of cement and aggregate, surface preparation of rock sample, age, and temperature, type of cracking and method of testing. Generally, bond strength increases with age, but not at the same rate as the cement paste itself. Bond strength was dependent primarily on age, but not on the temperature of curing. Bond strength developed more slowly than the paste strength, and that the bond strength was less sensitive than paste strength to variation in w/c ratio of the paste. Through this study, age largely effect the development of ITZ.

4.2 Aggregate Size Effect towards ITZ

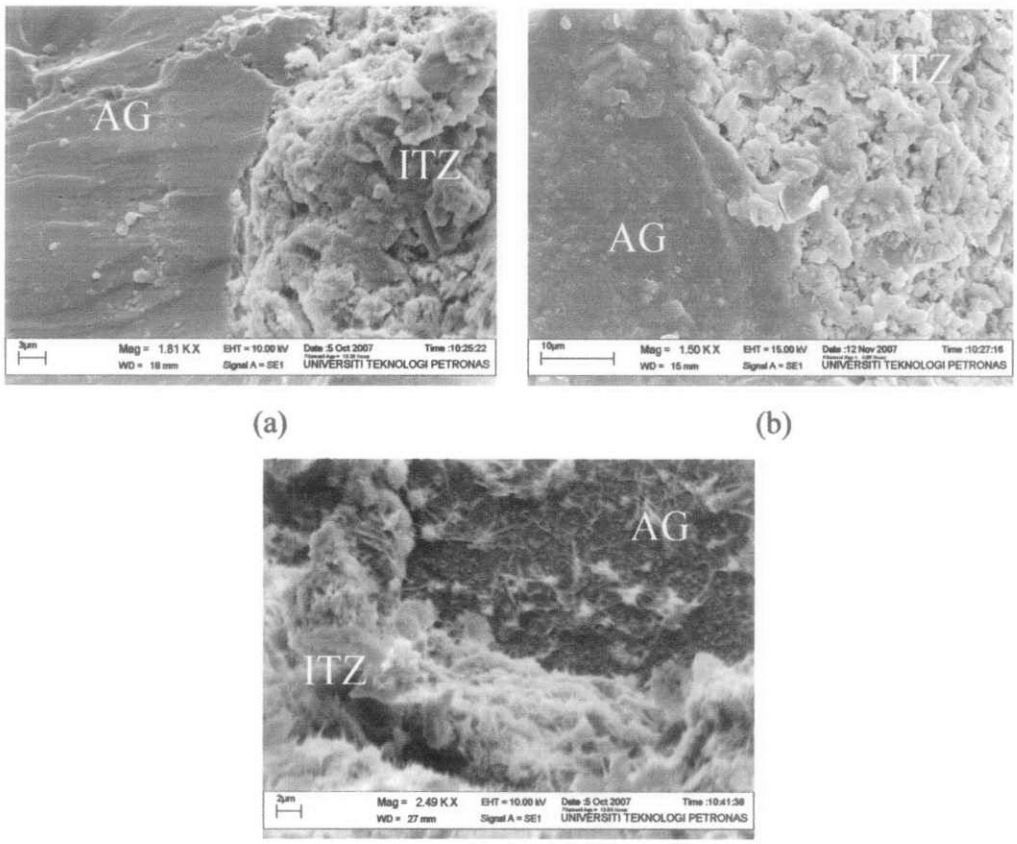
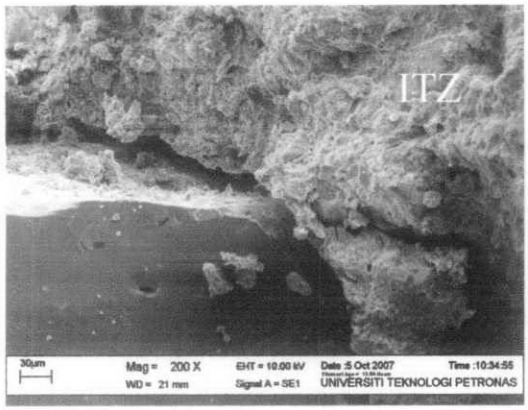
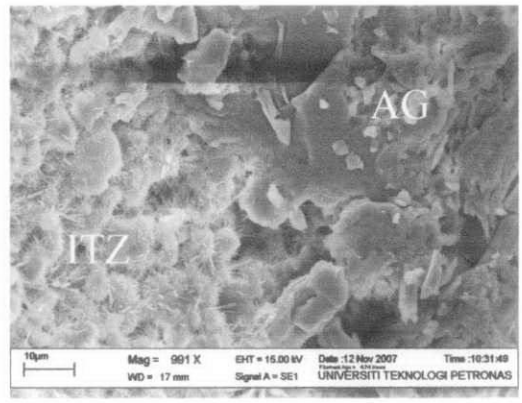


Figure 4.3: Microstructures view of ITZ (different size of aggregate). Aggregate marked by *AG*.(a) 10mm 3 days (b) 14mm 3 days (c) 20mm 3 days

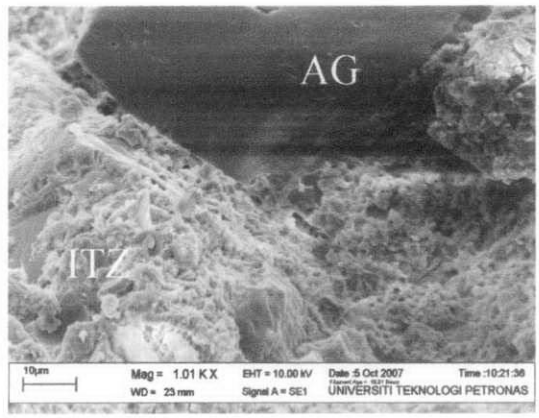
From Figure 4.3, by comparison among different aggregate size of 10, 14 and 20mm at 3 days, for 10 mm and 14 mm, there were still many unhydrated products can be seen. However, for 20 mm, hydration rate was faster by the presence of more needle-like C-S-H products and CH crystals at the aggregate surface. This may be because 20mm aggregate can contains more free water for hydration to happen and larger aggregate tends to form a continuous layer of water at the surface which would in turn favor the formation of larger CH crystals. The entire samples have high porosity, with 10mm sample seems to be worse, with bigger voids or pores.



(a)



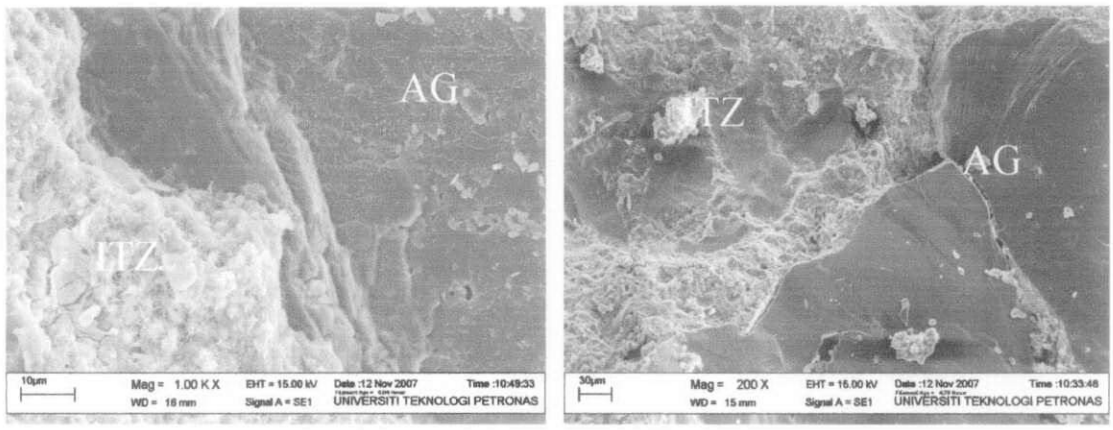
(b)



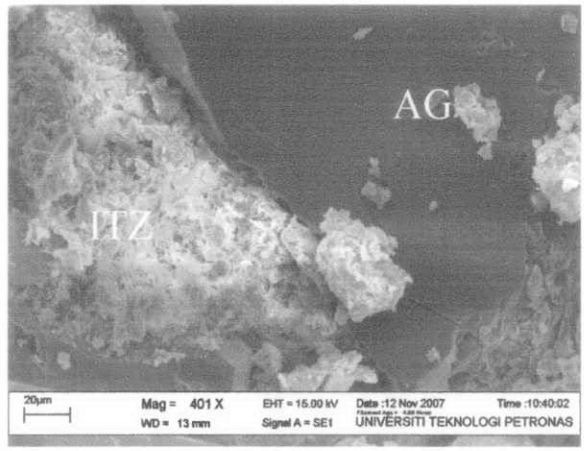
(c)

Figure 4.4: Microstructures view of ITZ at 7 days for different size of aggregate. Aggregate marked by *AG*. (a) 10mm 7 days (b) 14mm 7 days (c) 20mm 7 days

From Figure 4.4, for 10mm aggregate, the ITZ look loosen than the 14 and 20 mm aggregate which there is a very large gap between cement paste and the aggregate. However, high porosity still can be observed clearly for all three samples. More products of hydration can be seen in all the samples. In 14 mm, there are clearly more C-S-H needle shape products can be observed. This might explained why the tensile strength of 14mm is higher than both 10 mm and 20mm (See Table 4.1). In theoretically, smaller aggregate size should give better strength. Result of 10mm is smaller maybe due to its large surface area of aggregate available. Thus causes insufficient cement or water to react or hydrate around aggregate. To prevent this, lower w/c ratio need to be used for 10mm aggregate concrete mixing.



(a) (b)



(c)

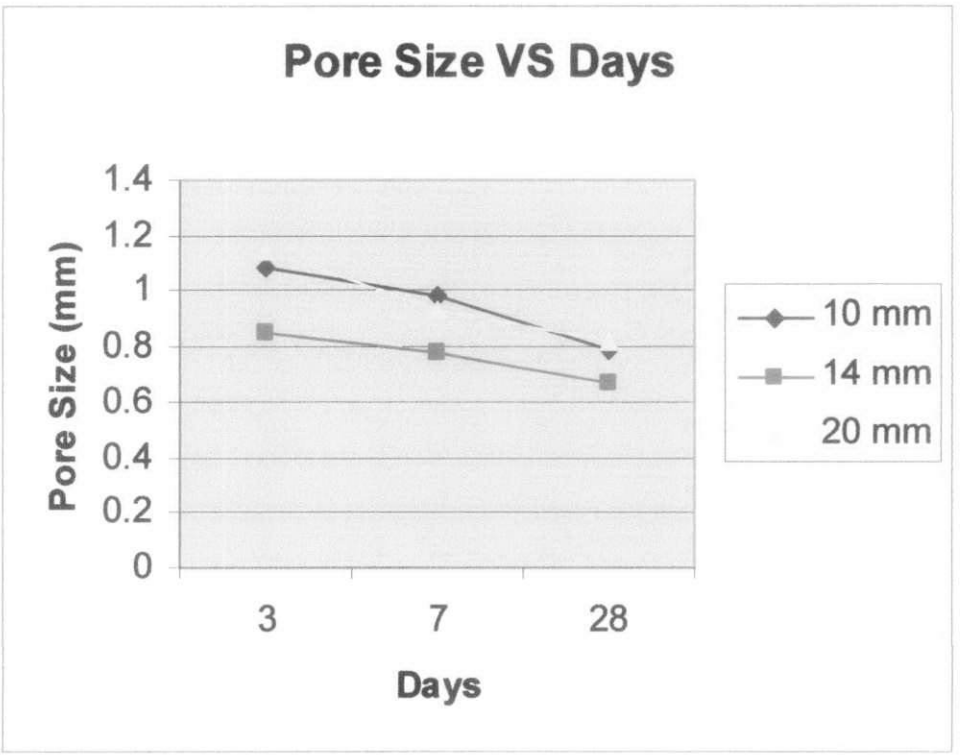
Figure 4.5: Microstructures view of ITZ. Aggregate marked by *AG.*
(a) 10mm 28 days (b) 14mm 28 days (c) 20mm 28 days

From Figure 4.5, the hydration products such as C-S-H and smaller CH crystals filled the void and pores. However, still many pores could be observed. Among the three samples, 10 mm and 14 mm structure look denser but the structure in 20 mm sample look quite loose with more pores than the others. This according to the result of pore size obtained (Table 4.1). On the 28 days, the pore size for 10mm becomes smaller than 20mm. With respect to age, the hydration will increase and the microstructures become denser. It can be concluded that size of aggregate does affect the characteristic of interfacial transition zone in concrete.

Table 4.1: Pore Size calculations and comparison

Size (mm)	Pore size (μm2)			Split tensile		
	3	7	28	3	7	28
10	1.081	0.986	0.796	4.10	5.44	8.50
14	0.851	0.785	0.667	6.68	8.43	11.00
20	1.148	0.936	0.82	4.40	6.62	10.35

Figure 4.6: Graph of pore size calculations



4.3 Experiment Results and Graphs

Table 4.2: Split Tensile

Aggregate 10mm

Days	3		7		28	
	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)
Cube 1	56.7	3.20	95.6	5.39	156.5	8.83
Cube 2	83.9	4.73	97.3	5.49	150.9	8.51
Cube 3	77.2	4.36	96.1	5.42	144.7	8.16
Average	72.6	4.10	96.3	5.44	150.70	8.50

Aggregate 14mm

Days	3		7		28	
	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)
Cube 1	123.4	6.96	146.2	8.25	196.3	11.08
Cube 2	113.3	6.39	152.5	8.60	193.7	10.93
Average	118.35	6.68	149.4	8.43	195.00	11.00

Aggregate 20mm

Days	3		7		28	
	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)	Max. Load (kN)	Stress (N/mm^2)
Cube 1	76.3	4.30	111.3	6.28	171.7	9.69
Cube 2	81.6	4.60	129.7	7.32	185.4	10.46
Cube 3	76.1	4.29	111.2	6.27	193	10.89
Average	78	4.40	117.4	6.62	183.37	10.35

Summary

	Stress (N/mm^2)		
Days	10mm	14mm	20mm
3	4.1	6.68	4.4
7	5.44	8.43	6.62
28	8.8	11	10.35

Table 4.3: Flexural Test

Aggregate 10mm

Sample	28 days	
	Max Load (kN)	Stress (N/mm^2)
Prism 1	9.32	6.99
Prism 2	10.51	7.88
Prism 3	10.44	7.83
Average	10.09	7.57

Aggregate 14mm

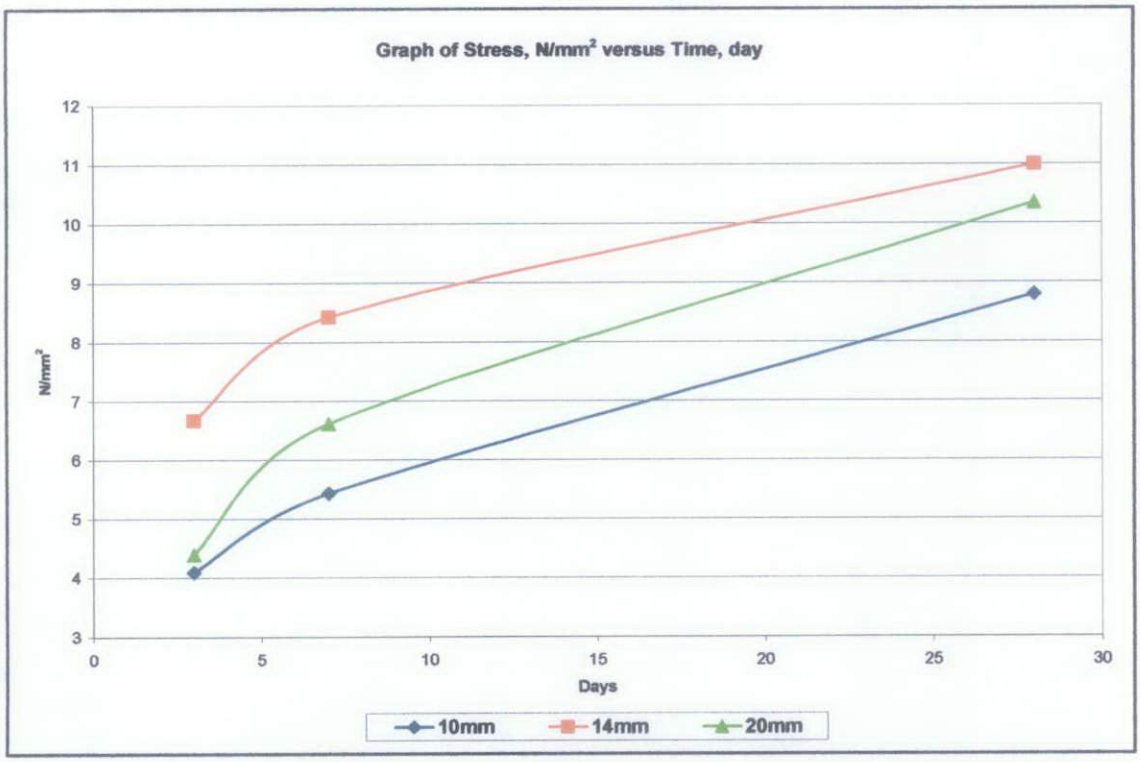
Sample	28 days	
	Max Load (kN)	Stress (N/mm^2)
Prism 1	12.81	9.6
Prism 2	11.27	6.76
Prism 3		
Average	12.04	8.18

Aggregate 20mm

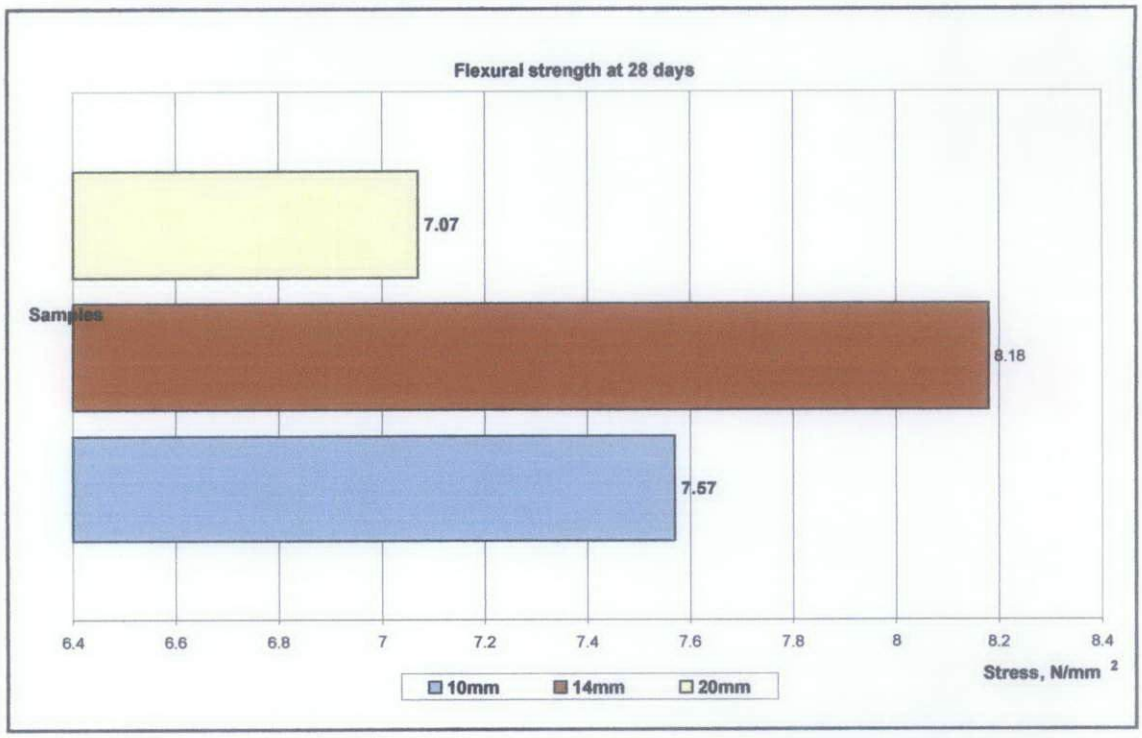
Sample	28 days	
	Max Load (kN)	Stress (N/mm^2)
Prism 1	9.49	7.12
Prism 2	9.43	7.07
Prism 3	9.35	7.01
Average	9.42	7.07

Summary

	Stress (N/mm^2)		
Days	10mm	14mm	20mm
28	7.57	8.18	7.07



(a)



(b)

Figure 4.7: Graphical result for tensile strength test.

(a) Split tensile test (b) Flexural test

Table 4.4 Compressive Strength Test

Aggregate 10mm

Sample	28 days	
	Max Load (kN)	Stress (N/mm^2)
Cube 1	335	33.5
Cube 2	320	32
Cube 3	332.4	33.24
Average	329.13	32.91

Aggregate 14mm

Sample	3		7		14	
	Max Load (kN)	Stress (N/mm^2)	Max Load (kN)	Stress (N/mm^2)	Max Load (kN)	Stress (N/mm^2)
Cube 1	324.7	32.47	343.1	34.31	467.5	46.75
Average	324.70	32.47	343.10	34.31	467.50	46.75

Aggregate 20mm

Sample	28 days	
	Max Load (kN)	Stress (N/mm^2)
Cube 1	449.4	44.95
Cube 2	403.2	40.32
Cube 3	445.2	44.52
Average	432.60	43.26

Summary

	Stress (N/mm^2)		
Days	10mm	14mm	20mm
3		32.47	
7		34.31	
28	32.91	46.75	43.26

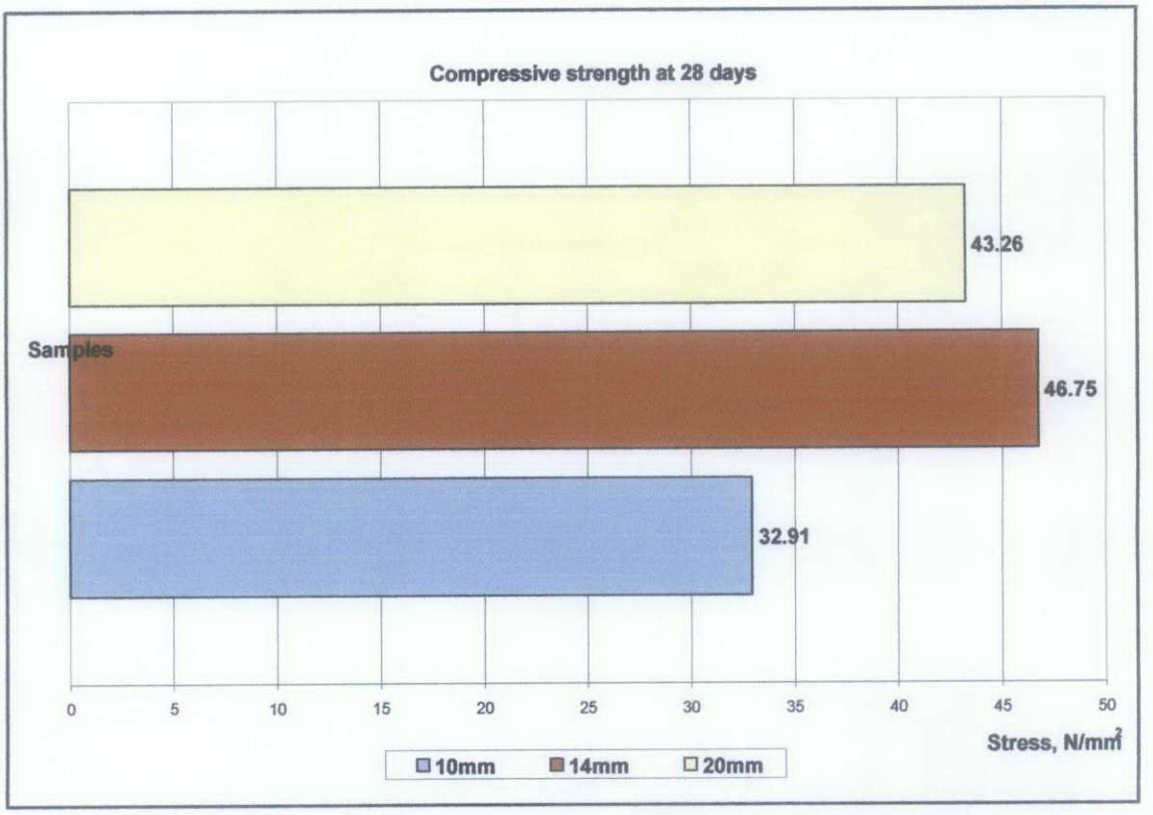


Figure 4.8: Graphical result for compressive strength test

CHAPTER 5

CONCLUSION AND RECOMMENDATION

This Final Year Project's concern is to study the effect of aggregate size towards ITZ and the influence of interfacial transition zone, the bond between cement paste and aggregate and the effect of aggregates towards tensile strength. Microstructure of interfacial transition zone in concrete is obtained and studied using SEM. It can be concluded ageing largely affected the ITZ formation, strength and mechanism. For 100 x 100 x 100 cubes and 100 x 100 x 500 prisms concrete mixing with w/c ratio of 0.5, 14mm aggregate concrete mixing gives higher value of tensile strength and the best result compared to the 10mm and 20mm.

The ITZ was observed to be the deciding factor for the tensile strength and played little role on the compressive strength. The tensile strength decreased as the aggregate size increased. At the same w/c ratio and age, reducing the aggregate size for example from 14 - 20 mm aggregate to 10mm and lower aggregate tends to reduce the porosity and increase the content of unhydrated particles in the region surrounding the aggregate.

For recommendation, there are several modification can be made to alter interfacial zone in order to improve concrete properties. Modifications that can be made are like densification of the interfacial transition zone, grain refinement and improving physico-chemical interaction between cement paste and aggregates. Densification can be achieved by using a very fine mineral admixture. Such fine powders appear to have four effects: (a) they improve the packing density in the interfacial zone, thereby largely eliminating wall effect. (b) They act to reduce bleeding, thus reduce the size of ITZ. (c) They act as growth nuclei for multiple generations of CH crystals which therefore have smaller size of crystals. (d) They participate in longer term pozzolanic reactions which continue to densify the ITZ with time. The grain refinement improvement can be achieved by using ASTM Type K (expansive) cement or using carbonate aggregates.

Type K cement replacing large CH crystals with small CH crystals having random orientation, and apparently leads to impoved concrete strength. Carbonate aggregates favors grain refinement by a process of dissolution of large CH crystals. Physico-chemical process often involve pre-treating or pre-activating aggregates with chemical solutions, or low w/c ratio pastes, thus supposedly giving them a greater affinity for reacting or interacting with the cement paste.

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